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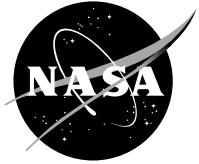
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STRENGTH, FRACTURE TOUGHNESS, AND SLOW CRACK GROWTH OF ZIRCONIA/ALUMINA COMPOSITES AT ELEVATED TEMPERATURE

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Various electrolyte materials for solid oxide fuel cells were fabricated by hot pressing 10 mol % yttria-stabilized zirconia (10-YSZ) reinforced with two different forms of alumina—particulates and platelets—each containing 0 to 30 mol % alumina. Flexure strength and fracture toughness of platelet composites were determined as a function of alumina content at 1000 °C in air and compared with those of particulate composites determined previously. In general, elevated-temperature strength and fracture toughness of both composite systems increased with increasing alumina content. For a given alumina content, flexure strength of particulate composites was greater than that of platelet composites at higher alumina contents (≥ 20 mol %), whereas, fracture toughness was greater in platelet composites than in particulate composites, regardless of alumina content. The results of slow crack growth (SCG) testing, determined at 1000 °C via dynamic fatigue testing for three different composites including 0 mol % (10-YSZ matrix), 30 mol % particulate and 30 mol % platelet composites, showed that susceptibility to SCG was greatest with SCG parameter $n = 6$ to 8 for both 0 mol and 30 mol % particulate composites and was least with $n = 33$ for the 30 mol % platelet composite.

INTRODUCTION

Solid oxide fuel cells (SOFC) are currently being developed for various power generation applications. The major components of a SOFC are the electrolyte, the anode, the cathode, and the interconnect. The two porous electrodes, anode and cathode, are separated by a fully dense solid electrolyte. Currently, yttria-stabilized zirconia (YSZ) is the most commonly used electrolyte in SOFC because of its high oxygen ion conductivity, stability in both oxidizing and reducing environments, availability, and low cost [1]. However, similar to other ceramics, YSZ is brittle and susceptible to fracture due to the existence of flaws, which are introduced during fabrication and use of the SOFC. In addition, the properties of YSZ such as low thermal conductivity and relatively high thermal-expansion coefficient make this material thermal-shock sensitive. Fracture in the solid oxide electrolyte will allow the fuel and oxidant to come in contact with each other resulting in reduced cell efficiency or in some cases malfunction of the SOFC. Therefore, YSZ solid electrolyte with high fracture toughness as well as enhanced strength is required from a performance and structural reliability point of view. Furthermore, because of high operating temperature (around 1000 °C) in SOFCs, it is certainly expected that slow crack growth would take place, which limits the life of electrolyte or SOFC system. Therefore, it is important to determine slow crack growth or life prediction parameters of the material to ensure accurate life prediction of SOFC components at elevated temperatures.

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The main objective of this work was to improve the strength and fracture toughness of the YSZ electrolyte for SOFC applications without adversely affecting its high-temperature ionic conductivity to an appreciable extent. The 10 mol % yttria-stabilized zirconia (10-YSZ) was reinforced with two different forms of alumina, particulates and platelets, each containing 0, 5, 10, 20, and 30 mol % alumina through mixing, milling, and hot pressing to full density [2,3]. Flexure strength and fracture toughness of YSZ/alumina composites were determined as a function of alumina content at ambient temperature for both particulate [2] and platelet [3] composites and at 1000 °C for the particulate composites [2], together with elastic modulus, density, and microhardness. Ambient-temperature flexure strength and fracture toughness of both particulate and platelet composites increased with increasing alumina content, reaching a maximum at 30 mol % alumina. For a given alumina content, flexure strength of the particulate composites was greater than that of the platelet composites, whereas, the difference in fracture toughness between the two composite systems was negligible. No virtual difference in elastic modulus and density was observed for a given alumina content between particulate and platelet composites.

This paper, as an extension of the previous work [2,3], describes behavior of flexure strength and fracture toughness of the platelets composites as a function of alumina content at 1000 °C, which is close to typical operating temperature of SOFC systems. The high-temperature flexure strength and fracture toughness of the platelet composites were compared with those of particulate composite determined previously [2]. Slow crack growth required for component design and life prediction was determined in flexure at 1000 °C in air using dynamic fatigue testing for selected materials including 0 mol % (10-YSZ matrix), 30 mol % particulate and 30 mol % platelet composites.

EXPERIMENTAL PROCEDURES

Processing and Test Specimen Preparations

Material processing was essentially the same as described elsewhere [2,3]. Briefly, the starting materials used were alumina powder [2] (high purity BAILALOX CR-30) from Baikowski International Corporation, Charlotte, NC, 10-mol % yttria fully stabilized zirconia powder (HSY-10) from Daiichi Kigenso Kagaku Kogyo Co., Japan, and alpha alumina hexagonal platelets (Pyrofine Plat Grade T2) [3] from Elf Atochem, France. Appropriate quantities of alumina and zirconia powders were slurry mixed in acetone and mixed for ~24 h using zirconia media. Acetone was then evaporated and the powder dried in an electric oven. The resulting powder was loaded into a graphite die and hot pressed at 1500 °C in vacuum under 30 MPa pressure into 152 by 152 mm billets using a large hot press. Grafoil was used as spacers between the specimen and the punches. Various hot pressing cycles were tried in order to optimize the hot pressing parameters that would result in dense and crack free ceramic samples. The processing flow diagram is shown in Figure 1. Five different YSZ/alumina composites containing 0 to 30 alumina mol % were fabricated for each of particulate and platelet composite systems.

The billets were machined into flexure test specimens with nominal depth, width and length of 3.0 by 4.0 by 50 mm, respectively, in accordance with ASTM test standard C 1211 [4]. Machining direction was longitudinal along the 50 mm-length direction. It should be noted that unlike transformation-toughened (from *tetragonal* to *monoclinic*) zirconias, the *cubic* yttria-stabilized zirconia is very unlikely to induce any transformation-associated residual stresses on the surfaces of test specimens due to machining. The sharp edges of test specimens were chamfered to reduce any spurious premature failure emanating from those sharp edges.

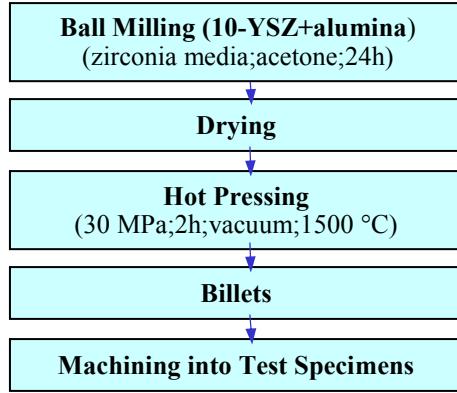


Figure 1. Processing flow diagram for 10-mol % yttria-stabilized zirconia/alumina composites, applied to both particulate and platelet composites [2,3].

Flexure Strength and Fracture Toughness Testing

Flexure strength was determined as a function of alumina content for the platelet composites at 1000 °C in air. Note that both flexure strength and fracture toughness of the particulate composites were determined previously at 1000 °C in air as a function of alumina content [2]. A SiC four-point flexure fixture with 20 mm-inner and 40 mm-outer spans was used in conjunction with an electromechanical test frame (Model 8562, Instron, Canton, MA). A fast test rate of 50 MPa/s was applied in load control to reduce slow crack growth effect of the materials.[‡] A total of 10 test specimens were tested for each alumina content. Testing was followed in accordance with ASTM test standards C 1211 [4].

Fracture toughness of the platelet composites using flexure bar specimens measuring 3 by 4 by 25 or 50 mm was determined at 1000 °C in air using single edge v-notched beam (SEVNB) method [6]. This method was verified as an appropriate technique at elevated temperature free from crack healing due to oxidation [2]. This method utilizes a razor blade with diamond paste with a grain size of 9 µm to introduce a final sharp notch with a root radius ranging 10 to 20 µm by tapering a saw notch [2,3,6]. The sharp v-notched specimens with a notch depth of 0.9 mm were fractured in a four-point flexure fixture with 20 (or 10) mm-inner and 40 (or 20) mm-outer spans using the electromechanical test frame (Model 8562, Instron) at an actuator speed of 0.5 mm/min. A total of five specimens were tested for each alumina content. Fracture toughness K_{IC} was calculated based on the formula by Srawley and Gross [7] as follows:

$$K_{IC} = \frac{P_f(L_o - L_i)}{BW^{3/2}} \frac{3\alpha^{1/2}}{2(1-\alpha)^{3/2}} f(\alpha) \quad (1)$$

where P_f , L_o , L_i , B , W are fracture load, outer span, inner span, specimen width, and specimen depth, respectively, $\alpha = a/W$ with a being precrack size, and $f(\alpha)$ is expressed

$$f(\alpha) = 1.9887 - 1.326\alpha - \frac{\alpha(1-\alpha)(3.49 - 0.68\alpha + 1.35\alpha^2)}{(1+\alpha)^2}$$

[‡] Elevated temperature strength of many advanced ceramics depends on test rate due to slow crack growth during testing. It has been shown that elevated-temperature strength of advanced ceramics increases with increasing test rate and converges to ambient-temperature strength (or inert strength) at “ultra”-fast test rates $\geq 10^5$ MPa/s [5]. Although the test rate of 50 MPa/s used in this work was not sufficient to obtain an appropriate “inert” strength whereby no slow crack growth occurs, the test rate of 50 MPa/s was chosen to determine the conventional, so-called “fast-fracture” strength of the material in which a test rate of around 30 to 100 MPa/s is typically employed.

Slow Crack Growth (Dynamic Fatigue) Testing

Slow crack growth (SCG) behavior of the chosen composites was determined at 1000 °C in air using dynamic fatigue (or called ‘constant stress-rate’) testing in accordance with ASTM test method C 1465 [8]. Three different composites including 0 and 30 mol % alumina particulate and 30 mol % alumina platelet composites were chosen based on the results of optimum strength and fracture toughness properties exhibited by the 30 mol % particulate and platelet composites [2,3]. The additional 0 mol % composite, 10-YSZ matrix, was used to generate the baseline data. Dynamic fatigue testing was performed in flexure using flexure test specimens (3 by 4 by 50 mm) at two different test rates of 50 and 0.005 MPa/s for a given composite under load control of the electromechanical test frame (Model 8562, Instron). A SiC four-point flexure fixture with 20/40 mm spans was used. A total of 10 test specimens were used at each test rate for a given composite.

RESULTS AND DISCUSSION

Flexure Strength

The results of elevated-temperature strength testing for the platelet composites are presented and compared with the particulate composite strength [2] in Figure 2, where average strength was plotted for clarity as a function of alumina mol % for both composite systems. The strength of the platelet composites, except for 10 mol %, remained almost unchanged or a little decreased from 10 mol % with increasing alumina content. The maximum strength occurring at 10 mol % was 17% greater than the ‘zero’ alumina-content strength. The strength of the platelet composites was greater at 5 and 10 mol % but lower at 20 and 30 mol % than the particulate composites counterpart. The trend in strength increase with alumina content was more significant in the particulate composites than in the platelet composites. The strength of the particulate composites with respect to the ‘zero’-alumina content (10-YSZ) strength decreased initially at 5 mol % and increased thereafter with increasing alumina content, reaching a maximum at 30 mol %. The maximum strength of the particulate composites occurring at 30 mol % alumina content was 40% greater than the ‘zero’-alumina content strength. Also, the particulate strength at 30 mol % was 30% greater than the respective platelet strength.

As also can be seen from Figure 2, the strength scatter was less significant in the platelet composites than in the particulate composites. Weibull modulus (m), estimated despite a limited number (10) of specimens tested at each alumina content, was in the range of $m = 8$ to 19 and $m = 5$ to 10, respectively, for the platelet and particulate composites. Fracture originated distinctly from surface-connected defects (“surface flaws”) for both composites. Platelets, mostly consistent in size, were frequently associated as primary failure origins. Hence, alumina platelets acted as strength-controlling flaws rather than as reinforcing media, typical of many platelets-reinforced composites, resulting in less scatter in strength or higher Weibull modulus. The reason why the particulate composites exhibited improved strength over the platelet composites particularly at 30 mol % content is primarily due to the fact that alumina particulates might have acted as a major source of reinforcement. The effect of residual stress on overall resulting strength of the platelet composites, due to CTE mismatches between matrix and platelets, was shown negligible from the previous thermal cycling experiment [3].

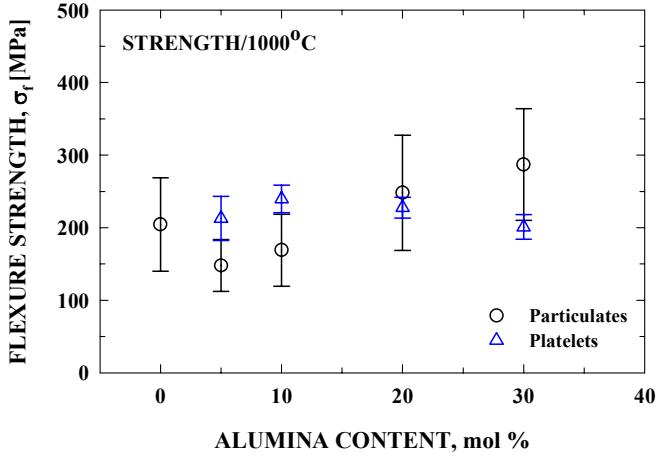


Figure 2. Flexure strength of 10-YSZ/alumina platelet composites as a function of alumina content at 1000 °C in air. Flexure strength of 10-YSZ/alumina particulate composites [2] is included for comparison. Error bars indicate ± 1.0 standard deviations.

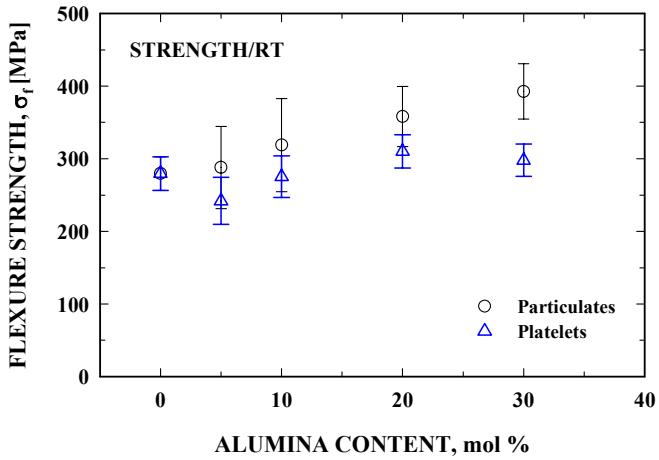


Figure 3. Flexure strength of 10-YSZ/alumina particulate and platelet composites as a function of alumina content at ambient temperature in air [2,3]. Error bars indicate ± 1.0 standard deviations.

For comparison, ambient-temperature flexure strength of both platelet and particulate composites determined previously [2,3] are presented in Figure 3. The particulate composite strength increased almost linearly with increasing alumina content with a maximum 40% increase at 30 mol %, while the platelet composite strength was not significantly dependent on alumina content with a maximum 11% increase at 20 mol %. Flexure strength of the particulate composites for a given alumina content was approximately 15% greater than that of the platelet composites counterpart. Similar to the case of elevated-temperature strength, the scatter of ambient-temperature strength was all greater in the particulate composites than in the platelet composites, as seen in Figure 3. More exactly, Weibull modulus was found as $m \approx 13$ and 7, respectively, for the particulate and platelet composites. The overall ambient-temperature strength of the two composite systems for a given alumina content was greater (20 to 50% and 10 to 30%, respectively, for the particulate and platelet composites) than their elevated-temperature counterpart, attributed to strength degradation at elevated temperature presumably by slow crack growth phenomenon.

Fracture Toughness

A summary of fracture toughness of the platelet composites determined at 1000 °C is presented in Figure 4, in which the values of fracture toughness by the SEVNB method were plotted as a function of alumina mol %. Fracture toughness of the particulate composites determined at 1000 °C in air [2] was also included for comparison. Fracture toughness of the platelet composites increased monotonically with increasing alumina content with an initial jump from 0 to 5 mol %, reaching a maximum at 30 mol %. Fracture toughness increased significantly by 74% when alumina content increased from 0 to 30 mol %. In a similar way, fracture toughness of the particulate composites, in general, increased with alumina content, resulting in a maximum increase of 50% at 30 mol % alumina. Unlike the elevated-temperature strength, the difference in fracture toughness between the particulate and platelet composites was distinct for all of alumina contents. Fracture toughness was greater (15 to 80%) in the platelet composites than in the particulate composite, irrespective of alumina content, indicating that toughening by addition of platelets to 10-YSZ matrix might have been in effect through possible mechanisms such as bridging and/or crack deflection. At 30 mol % alumina content where maximum fracture toughness was attained for both composite systems, fracture toughness of the platelet composites was approximately 16% greater than that of the particulate composites.

It has been observed that an incompatibility is generally operative for many advanced monolithic ceramic composites between strength and fracture toughness in such a manner that one property increases while the other decreases. This seemed to be the case for the platelet composites that showed not only insensitive or somewhat decreasing strength but also fracture-toughness increase with increasing alumina content. This confirms again a notion that platelets were significantly effective in toughening but sacrificial in strengthening.

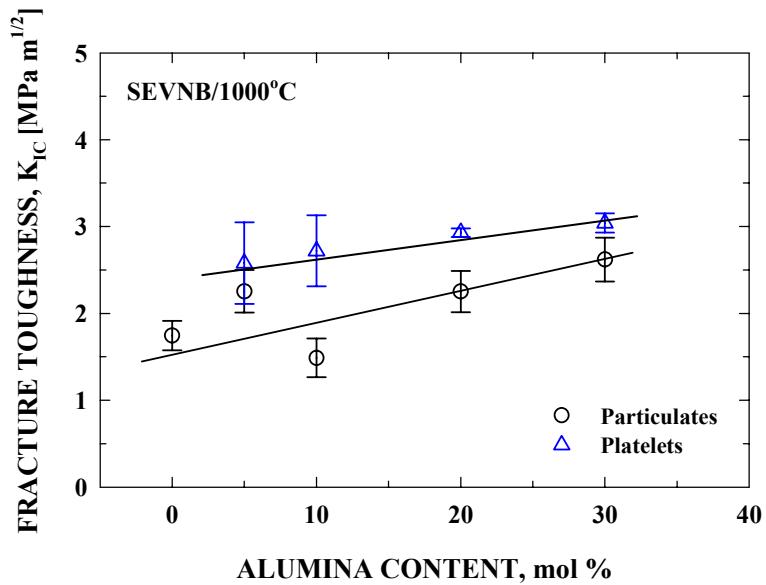


Figure 4. Fracture toughness of 10-YSZ/alumina platelet composites as a function of alumina content at 1000 °C in air. Fracture toughness of 10-YSZ/alumina particulate composites [2] is included for comparison. Error bars indicate ± 1.0 standard deviations. The lines represent the best fit.

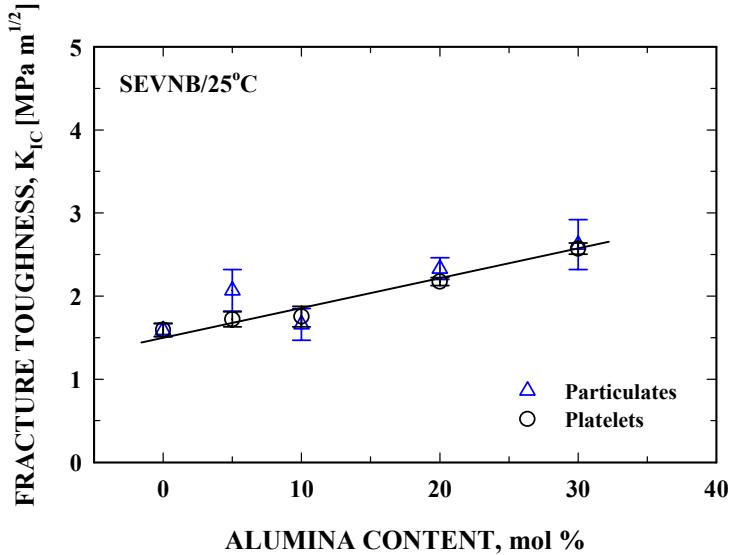


Figure 5. Fracture toughness of 10-YSZ/alumina platelet and particulate composites as a function of alumina content at ambient temperature (25 °C) in air [2,3]. Error bars indicate ± 1.0 standard deviations. The line represents the best fit for both particulate and platelet composites.

Fracture toughness of the two composite systems determined at ambient temperature [2,3] is presented for comparison in Figure 5. Fracture toughness increased with increasing alumina content for both composites, resulting in a maximum increase of about 60% at 30 mol % [2,3]. Contrast to the elevated-temperature fracture toughness, ambient-temperature fracture toughness exhibited little difference between the particulate and platelet composites. It is also noted that the difference in fracture toughness of the particulate composites between 25 and 1000 °C was negligible, as can be seen by comparing Figures 4 and 5. Therefore, fracture toughness behavior of the platelet composites at 1000 °C is unique and therefore imposes a difficulty in terms of understanding governing mechanisms, since the platelet composites yielded much higher fracture toughness at elevated temperature than at ambient temperature. The only plausible explanation is that toughening mechanism(s) in the platelet composites might have been more operative at elevated temperature together with occurrence of possible crack healing and/or crack-tip plasticity. A further detailed fractographic analysis is a prerequisite to pinpoint governing mechanism(s) associated.

Slow Crack Growth: Dynamic Fatigue

The results of slow crack growth ('dynamic fatigue' or 'constant stress-rate') testing at 1000 °C in air for three different composites including 0 mol % (10-YSZ matrix) and 30 mol % particulate and 30 mol % platelet composites are shown in Figure 6, where fracture stress of each composite was plotted as a function of applied stress rate. The decrease in fracture stress with decreasing stress rate, which represents susceptibility to slow crack growth (SCG), was evident for all three composites with its degree of strength degradation with decreasing stress rate being dependent on material. The basic underlying formulation of slow crack growth for many advanced monolithic ceramics and composites (reinforced with particulates, platelets or whiskers) at elevated temperatures follows the following power-law form [9]

$$v = A[K_I / K_{IC}]^n \quad (2)$$

where v , K_I and K_{IC} are crack velocity, mode I stress intensity factor, and mode I fracture toughness, respectively. A and n are material/environment dependent SCG parameters. The responsible mechanism of SCG at elevated temperatures has been known as grain boundary sliding. In case of dynamic fatigue

loading, a constant stress rate ($\dot{\sigma}$) is applied to a test specimen until the test specimen fails. The corresponding fracture stress (σ_f) can be derived from Eq. (2) and related stress intensity factor with some mathematical manipulations to give [8, 10,11]

$$\log \sigma_f = \frac{1}{n+1} \log \dot{\sigma} + \log D \quad (3)$$

where D is another SCG parameter associated with A , n , K_{IC} , inert strength, and crack geometry factor [8,11]. The SCG parameters n and D can be determined from the slope and intercept by a linear regression analysis when \log (*fracture stress*) is plotted as a function of \log (*applied stress rate*). Equation (2) is the basis commonly used in dynamic fatigue testing, which has been adopted to determine SCG parameters of advanced ceramics in ASTM test standards at both ambient and elevated temperatures as well [8,11].

The results shown in Figure 6 were plotted according to Eq. (2) with units of MPa for σ_f and MPa/s for $\dot{\sigma}$. The SCG parameters n and D were found to be $n = 8$ and $D = 126$, $n = 6$ and $D = 151$, $n = 33$, and $D = 179$, respectively, for 0 mol % (10-YSZ), 30 mol % particulate and 30 mol % platelet composites, as summarized in Table 1. The respective correlation coefficients (r_{coef}) of the linear regression lines were $r_{\text{coef}} = 0.8226$, 0.9558 , and 0.8860 . Both 0 mol % and 30 mol % particulate composites exhibited very high susceptibility to SCG with relatively low SCG parameter $n = 6$ to 8 and the 30 mol % platelet composite exhibited intermediate susceptibility with $n = 33$.[§] Hence, the 30 mol % platelet composite exhibited higher resistance to SCG as compared with both 0 and 30 mol % particulate composites. The addition of 30 mol % alumina platelets into 10-YSZ matrix might have resulted in increased resistance to grain boundary sliding, while the addition of 30 mol % fine alumina particulates would not have had any positive effect on reducing or minimizing such grain boundary sliding. Note that the value of SCG parameter n determined at 1000 °C in air for typical aluminas with 96 to 99% purity is in a range of $n = 7$ to 13 [12,13]. Significant improvement in SCG resistance was obtained by the composite approach with two specific materials—YSZ and alumina (platelets)—in which each of YSZ and alumina exhibits a significantly high SCG susceptibility ($n = 6$ to 13) if they are used individually in a form of monolith.

Table 1. Summary of slow crack growth parameters determined for different composites via dynamic fatigue testing in flexure at 1000 °C

Materials	Applied stress rate (MPa/s)	No. of test specimens	Mean flexure strength (MPa)	Slow crack growth parameters	
				n	D
10-YSZ (matrix)	50 0.005	10 10	204.5(64.5)* 74.8(25.4)	8.3	126.3
10-YSZ/30 mol %-alumina particulate composite	50 0.005	10 10	287.0(77.0) 66.7(7.7)	5.5	150.6
10-YSZ/30 mol %-alumina platelet composite	50 0.005	10 10	201.1(17.1) 152.9(8.0)	32.9	178.5

Notes: Tests conducted in four-point flexure with 20/40 mm spans, ASTM C1465 [8] in ambient air; The number in the parenthesis indicates ± 1.0 standard deviation.

[§] Note that susceptibility to slow crack growth is typically categorized in advanced ceramics such that SCG susceptibility is very high for $n < 20$, intermediate for $n = 30$ to 50 , and very low for $n > 50$.

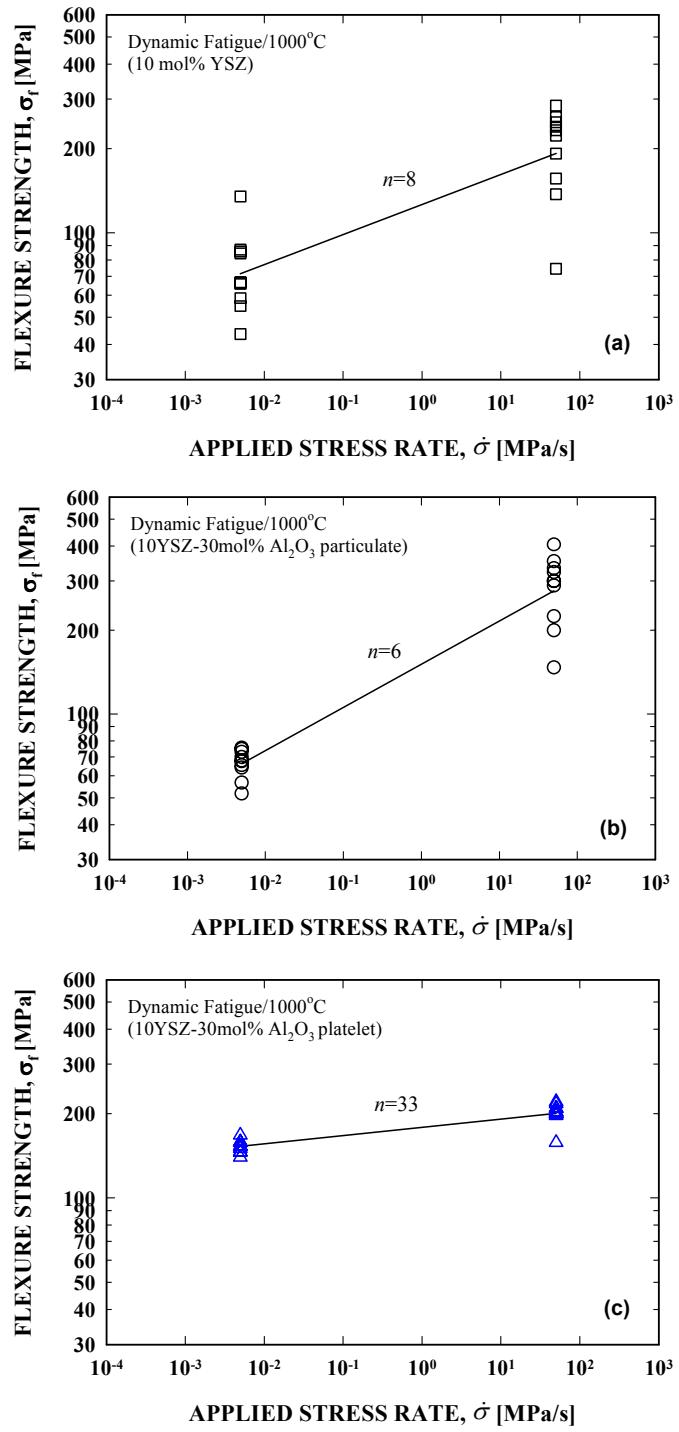


Figure 6. Results of dynamic fatigue ('slow crack growth' or 'constant stress-rate') testing in flexure for three different 10-YSZ/alumina composites at 1000 °C in air: (a) 10-YSZ (matrix); (b) 10-YSZ/30 mol % alumina particulate composite; (c) 10-YSZ/30 mol % alumina platelet composite.

The line indicates the best-fit based on Eq. (3).

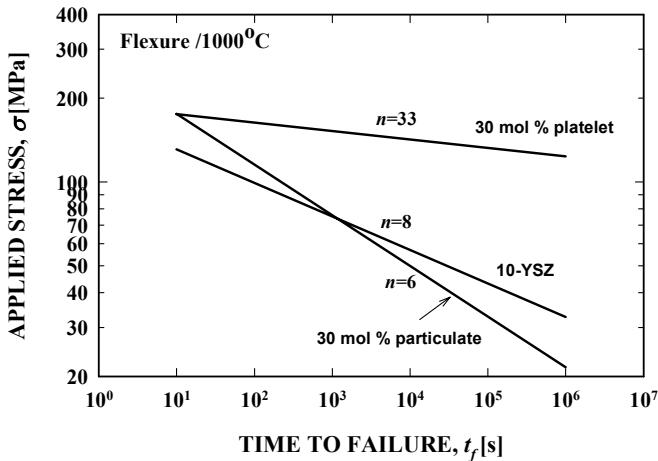


Figure 7. Life prediction diagram constructed from the dynamic fatigue results at 1000 °C for three different 10-YSZ/alumina composites including 10-YSZ (matrix), 10-YSZ/30 mol % alumina particulate composite, and 10-YSZ/30 mol % alumina platelet composite. The prediction represents at a failure probability of approximately 50%.

A life prediction diagram would give a better interpretation of SCG behavior among the three composites, which was constructed in Figure 7 under the same specimen configuration based on the following relation [14]

$$t_f = \left[\frac{D^{n+1}}{n+1} \right] \sigma^{-n} \quad (4)$$

where t_f and σ are time to failure (in sec) and constant applied stress (in MPa), respectively. Of course, the prediction is valid when the same failure mechanism(s) is operative, irrespective of loading condition, either dynamic or static. As can be seen from the figure, for a given applied stress lifetime is greatest and least, respectively, for the 30 mol % platelet composite and the 10-YSZ and 30 mol % particulate composite. As a consequence, the 30 mol % platelet composite would be a most reasonable choice among the three materials in conjunction with component life. A detailed life prediction and reliability of actual, complex fuel cell components can be made using analytical (finite element modeling) and reliability tool such as CARES/Life integrated computer code [15] with known SCG parameters of a material.

Choice of Material in View of Structural Reliability/Life of SOFC

As seen in the forgoing results, elevated-temperature flexure strength increased with increasing alumina content particularly at 30 mol % alumina for the particulate composites, but increased a little or remained almost unchanged with alumina content for the platelet composites. Fracture toughness increased with alumina content for both composite systems reaching a maximum at 30 mol %. At 30 mol % alumina content where both particulate and platelet composites exhibited both maximum flexure strength and fracture toughness, flexure strength of the particulate composites was 30% greater than that of the platelet composites, while fracture toughness of the platelets composites was 16% greater than the particulate composites. The resistance to SCG susceptibility was greater in the 30 mol % platelet composite with a higher SCG parameter of $n = 33$ than in the 30 mol % particulate composites with a lower SCG parameter of $n = 6$.

From the structural reliability/life point of view, a composite which gives long life and is strongest (in strength), toughest (in fracture toughness), stiffest (in elastic modulus) and lightest (in weight) is certainly of the best choice for fuel cells material. Elastic modulus was found increased with increasing alumina and density, by contrast, decreased with increasing alumina content [2,3]. It has been shown that the 30 mol % particulate composite was the best choice of material based on strength, fracture toughness, elastic modulus and density that were evaluated at *ambient temperature* [2,3]. Since operating temperatures of typical SOFCs are within or close to 1000 °C, a choice of a candidate material should not be solely based on ambient temperature properties but based on elevated temperature properties, particularly including slow crack growth which controls life of fuel cell components.

With respect to elevated-temperature strength, the particulate composite is better than the platelet counterpart. By contrast, with regard to fracture toughness and SCG resistance, the platelet composite is better than the particulate counterpart. Hence, a unified choice to satisfy all the important requirements—strong, tough, long life—can hardly be made. A case-by-case selection, depending on the types of operation/service conditions (temperature, loading (continuous or intermittent), environment, and components configurations, etc.), is needed. For example, if the components are subjected to a continuous, isothermal type of operations without frequent interruption (thus encountering little thermal shock loading, etc.), then the 30 mol % platelet composite would be a good candidate since the material would give longer service life of components. This structural consideration should not neglect the SOFCs' important electrical performance, that is, oxygen (O^2)-ion conductivity. Preliminary results, however, have shown that electrical conductivity of both composite systems was almost independent of alumina content [3].

SUMMARY AND CONCLUSIONS

1. The 10-mol % yttria-stabilized zirconia (10-YSZ)/alumina composites reinforced with alumina platelets containing 0 to 30 mol % alumina were tested at 1000 °C in air to determine flexure strength and fracture toughness. Slow crack growth was also determined in flexure at 1000 °C in air using dynamic fatigue testing for selected composites including 0 mol % (10-YSZ matrix) and 30 mol % particulate and 30 mol % platelet composites.
2. Elevated-temperature flexure strength of the platelet composites, except for 10 mol % alumina content, remained virtually unchanged with respect to the 0 mol % (10-YSZ matrix) strength. The strength of 10 mol % alumina composite was increased by 17%. However, flexure strength was approximately 30% lower in the platelet than in the particulate composites at 30 mol % where a maximum strength yielded for the particulate composites.
3. Elevated-temperature fracture toughness of the platelet composites, determined by SEVNB method, increased with increasing alumina content, reaching a maximum at 30 mol %. A 74% increase in fracture toughness was achieved when alumina content increased from 0 to 30 mol %. Fracture toughness was approximately 16% greater in the platelet composites than in the particulate composites.
4. The susceptibility to slow crack growth was high for both 0 and 30 mol % particulate composites with lower SCG parameters of $n = 6$ to 8, whereas the susceptibility to SCG was low for the 30 mol % platelet composite with its higher SCG parameter of $n = 33$. The resistance to SCG was thus significantly improved with the addition of 30 mol % platelet alumina into 10-YSZ matrix, which is very desirable for longer service lives of SOFC components.

REFERENCES

1. N.Q. Minh, "Ceramic Fuel Cells," *J. Am. Ceram. Soc.*, **76**[3] 563–588 (1993).
2. S.R. Choi and N.P. Bansal, "Strength and Fracture Toughness of Zirconia/Alumina Composites for Solid Oxide Fuel Cells," *Ceram. Eng. Sci. Proc.*, **23**[3] 741–750 (2002).
3. S.R. Choi and N.P. Bansal, "Processing and Mechanical Properties of Various Zirconia/Alumina Composites for Fuel Cells Applications," NASA/TM—2002-211580, National Aeronautics and Space Administration, Glenn Research Center, Cleveland, OH (2002); also presented at CIMTEC 2002 Conference, paper # G1:P03 (to be published in the proceedings), June 14–18, 2002, Florence, Italy.
4. ASTM C 1211 "Test Method for Flexural Strength of Advanced Ceramics at Elevated Temperatures," *Annual Book of ASTM Standards*, Vol. 15.01, American Society for Testing & Materials, West Conshohocken, PA (2001).
5. S.R. Choi and J.P. Gyekenyesi, "'Ultra'-Fast Fracture Strength of Advanced Structural Ceramics at Elevated Temperatures: An Approach to High-Temperature 'Inert' Strength," pp. 27–46 in *Fracture Mechanics of Ceramics*, Vol. 13, Edited by R.C. Bradt, D. Munz, M. Sakai, V. Ya. Shevchenko, and K.W. White, Kluwer Academic/Plenum Publishers, New York (2002).
6. J. Kübler, (a) "Fracture Toughness of Ceramics Using the SEVNB Method: Preliminary Results," *Ceram. Eng. Sci. Proc.*, **18**[4] 155–162 (1997); (b) "Fracture Toughness of Ceramics Using the SEVNB Method; Round Robin," VAMAS Report No. 37, EMPA, Swiss Federal Laboratories for Materials Testing & Research, Dübendorf, Switzerland (1999).
7. J.E. Srawley and B. Gross, "Side-Cracked Plates Subjected to Combined Direct and Bending Forces," pp. 559–579 in *Cracks and Fracture*, ASTM STP 601, American Society for Testing and Materials, Philadelphia (1976).
8. ASTM C 1465, "Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Elevated Temperatures," *Annual Book of ASTM Standards*, Vol. 15.01, American Society for Testing and Materials, West Conshohocken, PA (2001).
9. S.M. Wiederhorn, "Subcritical Crack Growth in Ceramics," pp. 613–646 in *Fracture Mechanics of Ceramics*, Vol. 2, Edited by R.C. Bradt, D.P.H. Hasselman, and F.F. Lange, Plenum Press, New York (1974).
10. A.G. Evans, "Slow Crack Growth in Brittle Materials under Dynamic Loading Condition," *Int. J. Fracture*, **10** [2] 251–259 (1974).
11. ASTM C 1368, "Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Ambient Temperature," *Annual Book of ASTM Standards*, Vol. 15.01, American Society for Testing and Materials, West Conshohocken, PA (2001).
12. K. Jakus, T. Service, and J.E. Ritter, "High-Temperature Fatigue Behavior of Polycrystalline Alumina," *J. Am. Ceram. Soc.*, **63**[1–2] 4–7 (1980).
13. S.R. Choi and J.P. Gyekenyesi, "Specimen Geometry Effect on the Determination of Slow Crack Growth Parameters of Advanced Ceramics in Constant Flexural Stress-Rate Testing at Elevated Temperatures," *Ceram. Eng. Sci. Proc.*, **20**[3] 525–534 (1999).
14. J.E. Ritter, "Engineering Design and Fatigue of Brittle Materials," pp. 667–686 in *Fracture Mechanics of Ceramics*, Vol. 4, Edited by R.C. Bradt, D.P.H. Hasselman, and F.F. Lange, Plenum Publishing Co., New York (1978).
15. N.N. Nemeth, L.M. Powers, L.A. Janosik, and J.P. Gyekenyesi, "Time Dependent Reliability Analysis of Monolithic ceramic Components using the CARES/LIFE Integrated Design program," pp. 390–408 in *Life Prediction Methodologies and Data for Ceramic Materials*, ASTM STP 1201, Edited by C.R. Brinkman and S.F. Duffy, American Society for Testing and Materials, Philadelphia (1994).

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Various electrolyte materials for solid oxide fuel cells were fabricated by hot pressing 10 mol % yttria-stabilized zirconia (10-YSZ) reinforced with two different forms of alumina—particulates and platelets—each containing 0 to 30 mol % alumina. Flexure strength and fracture toughness of platelet composites were determined as a function of alumina content at 1000 °C in air and compared with those of particulate composites determined previously. In general, elevated-temperature strength and fracture toughness of both composite systems increased with increasing alumina content. For a given alumina content, flexure strength of particulate composites was greater than that of platelet composites at higher alumina contents (≥ 20 mol %), whereas, fracture toughness was greater in platelet composites than in particulate composites, regardless of alumina content. The results of slow crack growth (SCG) testing, determined at 1000 °C via dynamic fatigue testing for three different composites including 0 mol % (10-YSZ matrix), 30 mol % particulate and 30 mol % platelet composites, showed that susceptibility to SCG was greatest with SCG parameter $n = 6$ to 8 for both 0 and 30 mol % particulate composites and was least with $n = 33$ for the 30 mol % platelet composite.			
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